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(54) Title: HYDRAULICALLY ENTANGLED WET LAID BASE SHEETS FOR WIPERS (57) Abstract A cloth-like nonwoven material useful for wiping and having strength, toughness, abrasion resistance and resistance to cer- tain solvents and/or chemicals is made from mixtures of wood pulp and staple fibers randomly distributed and hydraulically en- tangled with each other to form a coherent entangled fibrous structure having a thickness index of at least about 0.008 and a ratio of machine direction strength to cross machine direction strength of at least about 1.5.		

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HYDRAULICALLY ENTANGLED WET LAID BASE SHEETS FOR WIPERS .

FIELD OF THE INVENTION

The field of the present invention includes nonwoven composite materials, for example hydroentangled materials containing mixtures of wood pulp fibers and staple fibers, which may be used as wipers for industrial and other applications.

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BACKGROUND OF THE INVENTION

Nonwoven materials such as, for example, meltblown or spunbonded polypropylene may be used as wipers. In certain applications such as automobile finishing the wiper is usually moistened with one or more volatile or semi-volatile solvents such as, for example, isopropyl alcohol/water, n-heptane, naphtha, and C₅ to C₇ aliphatic hydrocarbons in order to remove grease, fingerprints and/or smudges from the automobile finish before painting or priming. Some solvents and/or other chemicals cause some components such as, for example, low molecular weight polyolefins to leach out onto the wiped surface rendering that surface unsuitable for painting. Many nonwoven materials are hydrophobic and require treatment with one or more surfactants to become wettable. The surfactant may also be transferred to the wiped surface rendering that surface unsuitable for painting or priming.

Some nonwoven materials have a low tendency to shed fibers and may be used as wipers in applications where lint and dust are undesirable such as, for example, micro-electronic manufacturing clean rooms. However, such wipes are typically treated with surfactants to provide the absorbance and clean wiping characteristics desired in such applications. Surfactant treatments typically comprise an anionic surfactant such as, for example sodium dioctyl sulfosuccinate which has a high metallic ion content. These metallic ions provide special problems since, if present in sufficient concentrations, they may adversely affect the electrical properties of metal oxide semiconductor.

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Additionally, certain nonwoven materials have a slow rate of electrical charge dissipation which results in static build-up. Static build-up on a wiper may cause problems such as, for example, discomfort for the user, hazards with flammable solvents or damage to sensitive electronic equipment.

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Nonwoven materials used in wiping applications typically require some bonding to maintain the integrity of the nonwoven web. Thermal bonding can reduce the content of "active" fibers available for absorption. Thermal bonding also results in a
10 stiffer material which may scratch or abrade a soft surface such as newly applied paint. Chemical bonding offers potential problems with extractable bonding agents.

Nonwoven materials such as, for example, bonded carded webs and
15 air laid webs can be hydroentangled into a coherent web structure and used as wipers. However, these materials typically have high strength in only one direction because the fibers in the web are oriented in only one direction during the initial web forming process. That is, the materials have high strength in one
20 direction such as, for example, the machine direction and relatively low strength in the cross machine direction. This inequality of strength is undesirable because the material is more likely to tear in the weak direction and because the material must be much stronger than necessary in one direction in
25 order to meet minimum strength requirements in the weak direction.

Composite hydroentangled materials containing staple fibers and wood pulp fibers are typically made by overlaying a wood pulp
30 tissue layer on a staple fiber web and hydraulically entangling the two layers. Each side of the resulting hydroentangled material usually has a noticeably different level of abrasion resistance from the other side because of the way the material is produced.

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- Wood pulp and combinations of wood pulp and staple fibers can be processed to make paper tissue and paper items which may be used as wipers. Although these wipers have desirable absorbency, economy, and resistance to certain solvents and chemicals, they generally have low strength (particularly when wet), low toughness, low abrasion resistance and undesirable levels of lint. Such wipers also have poor visual and tactile aesthetics. For example, these materials are typically thin and sheet-like having a thickness index of about 0.01 or typically less than 0.01. Some physical properties of these materials such as, for example, strength and abrasion resistance may be improved by adding binders. However, binders increase the cost of the wiper and may leave residue on the surface to be wiped.
- Wipers may also be formed from woven materials. Depending on the material used, the wipers may have desirable absorbency and strength but typically are expensive and must be reused in order to be economical. Reusable cloths are not desirable because they may retain foreign, possibly injurious objects from previous uses. Cloth made from natural fibers has the disadvantage that many natural fibers such as, for example, cotton have natural oils such as, for example, cotton oil that can be extracted by some solvents and deposited onto the wiped surface. Cloth made from man-made fibers such as, for example, polyester may not be able to absorb water unless the fibers are treated with a surfactant so that the fibers are wettable. The presence of surfactants is undesirable for the reasons noted above.

DEFINITIONS

- The term "Peak Load" as used herein is defined as the maximum amount of load or force encountered in elongating a material to break. Peak Load is expressed in units of force, i.e., gf.

The term "Peak Energy Absorbed" (Peak EA) as used herein is defined as the area under a load versus elongation (stress versus strain) curve up to the point of "peak" or maximum load. Peak EA is expressed in units of work, i.e., kg-mm.

- 5 The term "Total Energy Absorbed" (TEA) as used herein is defined as the total area under a load versus elongation (stress versus strain) curve up to the point where the material breaks. TEA is expressed in units of work, i.e., kg-mm.
- 10 The term "Peak Percentage Elongation" as used herein is defined as relative increase in length of a specimen when a material is extended to up to the point of "peak" or maximum load. Peak percentage elongation is expressed as a percentage of the original length of the material, i.e., [(increase in
- 15 length)/(original length)] X 100.

The term "Total Percentage Elongation" as used herein is defined as the relative increase in length of a specimen when a material is extended to up to the point where the material breaks. Total

20 percentage elongation is expressed as a percentage of the original length of the material, i.e., [(increase in length)/(original length)] X 100.

The term "Thickness Index" as used herein is defined as the

25 value represented by the ratio of the thickness and the basis weight of a material where the thickness is described in millimeters (mm) and the basis weight is described in grams per square meter (gsm). For example, the thickness index may be expressed as follows:

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$$\text{Thickness Index} = [\text{thickness(mm)}/\text{basis weight(gsm)}]$$

The term "machine direction" as used herein is defined as the direction of travel of the forming surface onto which fibers are deposited during formation of composite nonwoven material.

The term "cross-machine direction" as used herein is defined as the direction which is perpendicular to the machine direction.

- 5 The term "Isotropic Strength Index" as used herein is defined as the value represented by the ratio of the peak load of a material in one direction such as, for example, the machine direction with the peak load of the material in the perpendicular direction, for example, the cross-machine direction. The index
10 is typically expressed as the ratio of the machine direction peak load with the cross-machine direction peak load. Materials usually have an index of greater than one (1) unless a comparison of peak load in a particular direction is specified. An isotropic strength index near one (1) indicates an isotropic
15 material. An isotropic strength index significantly greater than one (1) indicates an anisotropic material.

The term "staple fiber" as used herein refers to natural or synthetic fibers having an approximate average length of from
20 about 1 mm to about 24 mm, for example, from about 6 mm to about 15 mm, and an approximate denier of about 0.5 to about 3, for example, from about 0.7 to about 1.5 denier.

The term "Total Absorptive Capacity" as used herein refers to the
25 capacity of a material to absorb liquid and is related to the total amount of liquid held by a material at saturation. Total Absorptive Capacity is determined by measuring the increase in the weight of a material sample resulting from the absorption of a liquid and is expressed, in percent, as the weight of liquid
30 absorbed divided by the weight of the sample. That is, Total Absorptive Capacity = $\frac{[(\text{saturated sample weight} - \text{sample weight}) / \text{sample weight}] \times 100}{}$.

The term "Mop Up Capacity" as used herein refers to the capacity of a material to absorb liquid after the material has been saturated and wrung to simulate the multiple use of a wiper. The mop up capacity is related to the amount of liquid remaining in a material after liquid is removed from a saturated material by wringing. Mop up capacity is determined by measuring the difference between the saturated weight and the wrung out weight of a material sample and dividing that amount by the weight of the dry sample. It is expressed, in percent, as the weight of liquid removed from the sample by wringing divided by the weight of the dry sample. That is, $[(\text{saturated sample weight} - \text{wrung out sample weight}) / \text{weight of dry sample}] \times 100$.

SUMMARY OF THE INVENTION

The present invention addresses the above-discussed problems by providing cloth-like nonwoven materials made from mixtures of wood pulp fibers and staple fibers randomly distributed and hydraulically entangled with each other to form a coherent entangled fibrous structure having a thickness index of at least about 0.008 and a isotropic strength index of not greater than about 1.5.

The materials of the present invention are made in a two step process. The materials are formed by conventional wet-forming techniques using an inclined wire. The materials are then hydroentangled using conventional hydroentangling techniques at pressures ranging from about 500 to about 2000 pounds per square inch (psi) and at speeds ranging from about 20 to about 300 meters per minute to form a coherent web structure without the use of thermal or chemical bonding.

The wet-formed materials of the present invention contain randomly distributed mixtures of wood pulp fibers and staple fibers. Typical materials contain from about 50 to about 90 percent by weight staple fiber and from about 10 to about 50

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percent by weight wood pulp fibers. Materials may contain up to about 100 percent staple fibers. The cloth-like nonwoven materials of the present invention have basis weights from about 30 to about 150 gsm.

- 5 Staple fibers used in the invention may have a denier in the range of about 0.7 to about 3 and an average length in the range of about 5 mm to about 18 mm. The staple fibers may be one or more of rayon, cotton, polyester, polyamides and polyolefins such as, for example, one or more of polyethylene, polypropylene, 10 polybutene, ethylene copolymers, propylene copolymers and butene copolymers. Long fiber wood pulps such as hardwood pulps are also particularly useful. Mixtures of long fiber and short fiber wood pulps may also be used.

15 DETAILED DESCRIPTION OF THE INVENTION

In accordance with the present invention there is provided a cloth-like composite nonwoven material having strength, toughness, abrasion resistance, resistance to certain solvents, and good visual and tactile aesthetics.

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The cloth-like nonwoven material is made from a dispersion of wood pulp fibers and staple fibers which is formed into a layer of randomly distributed fibers on a foraminous surface by conventional wet-laying techniques using an inclined wire.

- 25 Exemplary wet-forming processes are described in, for example, U.S. Patent No. 2,414,833 to Osborne, the disclosure which is hereby incorporated by reference.

In the headbox of the wet-forming apparatus, the dispersion of 30 fibers may be dilute, for example, containing about 2.5 grams of dry fiber per liter of fiber and water mixture. The consistency of the uniform layer of fibers after formation on the foraminous surface may range from about 10 to about 30 weight percent fiber solids in water. For example, the consistency may be about 25

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percent by weight solids. The uniform layer of fibers may be transferred to a different surface for entangling. The entangling surface may be, for example, a wire screen of from about 35 to about 100 mesh. The entangled material may be transferred to another surface for patterning. Mesh size and/or the texture of the foraminous patterning surface can be varied to create different visual and tactile properties. A coarse mesh such as, for example, from about 14 to about 35 mesh can be used to impart a textile or cloth-like appearance and feel.

10 The newly formed layer of randomly distributed fibers is hydraulically entangled to form a nonwoven material. Exemplary hydraulic entangling processes are described in, for example, U.S. Patent No. 3,485,706 to Evans, the disclosure of which is hereby incorporated by reference. For example, entangling may be
15 effected with a manifold produced by Honeycomb Systems, Incorporated containing a strip having 0.005 inch diameter orifices, 40 holes per inch and 1 row of holes. Other manifold configurations may also be used. The wet-formed materials may be run under the strip at speeds ranging from about 20 to about 300
20 meters per minute to be entangled by jets of liquid at pressures ranging from about 500 to about 2000 psi. It has been found that greater strength materials have been obtained by hydroentangling the base sheets at slower speeds and/or higher pressures. Additional passes through the hydroentangling equipment also
25 yields improved strength.

Patterning may be accomplished by transferring the entangled material to a coarse mesh such as, for example, 14 to about 35 mesh and running the material under the hydraulic entangling
30 apparatus at pressures from about 200 to about 1000 psi.

The nonwoven material formed by hydraulic entangling may be dried utilizing one or more conventional drying methods such as, for example, forced air, vacuum, heat or pressure. The nonwoven

material may be dried on a foraminous surface such as, for example, a wire mesh. Alternatively, the nonwoven material may be dried on an un-textured surface by conventional drying methods. Materials dried on a foraminous surface are softer and more drapable than materials dried on an un-textured surface. 5 Additionally, materials dried on a foraminous surface can be expected to have lower peak loads but greater peak elongations than materials dried on an un-textured surface.

In connection with this description certain test procedures have 10 been employed to determine oil and water absorption capacity and rate, linting, abrasion resistance, static decay, drape stiffness, sodium ion concentration, level of extractables, peak load, peak energy absorbed, total energy absorbed, peak elongation, and total elongation.

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Lint tests were carried out using a ClimetTM particle counter model Cl-250 available from the Climet Instrument Company, Redlands, California. Test were conducted essentially in accordance with INDA Standard Test 160.0 - 83 with the following 20 changes: (1) the sample size was 6 inches X 6 inches; and (2) the background count was not determined for each individual specimen tested. This test employed a mechanical particle generator which applied bending, twisting and crushing forces to sample specimens. Samples were placed in machine direction alignment in 25 an enclosure and twisted through an angle of 150° for a distance of 4.2 inches at a rate of about 70 cycles per minute. The enclosure is connected by tubing to the particle counter which draws the particles to the counter at a rate of about 20 cubic feet per hour. The flow rate through the instrument sensor is 30 1.0 cubic feet per hour. Each count takes 36 seconds and represents the number of particles of the specified size in 0.01 cubic feet of air.

Grab Tensile Test were conducted essentially in accordance with Method 5100 of Federal Test Method Standard No. 191A, utilizing samples of the entangled material having a width of about 4 inches and a length of about 6 inches. The samples were held at opposite ends by a one (1) square inch gripping surface. The 5 samples were tested with an Intellect II Model tensile testing apparatus available from Thwing Albert and with an Instron Model 1122 Universal Testing Instrument, each having a 3 inch jaw span and a crosshead speed of about 12 inches per minute. Values for peak load, peak energy absorbed, peak percentage elongation, 10 total energy absorbed and total percentage elongation were determined.

The rate of electrical charge dissipation of the material was determined essentially in accordance with Method 4046 of Federal 15 Test Method Standard No. 101B. Test results were obtained with an Electro/TechTM Calibrated Electrostatic Charge Detector with High Voltage Sample Holder using rectangular samples measuring 5-1/2 inches X 3-1/2 inches.

20 The rate that the material absorbed oil was determined as follows: A sample measuring 300 mm in the cross-machine direction and about 150 mm in the machine direction was placed flat on the liquid surface of an oil bath containing SAE 20W/50 motor oil. A stopwatch was used to record the time for the 25 sample to completely wet-out, that is, total saturation of 99 percent of the surface area of the sample. Non absorbent streaks of the material are not acceptable under the definition of complete wet-out but non absorbent individual fibers are acceptable. The rate that the material absorbed water was 30 determined by the same procedures utilized for oil except that distilled water was used instead of oil.

The capacity of the material to absorb oil was determined as follows: A dry 15 cm X 30 cm standard felt available from the

British Paper and Board Industry Federation, London, England was submerged for at least 24 hours in an oil bath containing SAE 20W/50 motor oil. The weight of a 10 cm X 10 cm material sample was determined to the nearest 0.01 gram. The sample was then submerged in the oil bath over the piece of felt until the sample
5 was completely saturated (at least 1 minute). The felt and sample were removed and suspended over the bath until the observed drainage of oil from the sample was complete. i.e., when the sample assumed a single overall color or appearance. The drained sample was weighed to the nearest 0.01 gram and the total
10 absorptive capacity was calculated.

The mop up capacity of the material was determined from the sample in the total absorptive capacity test by folding the saturated sample in half, and then in half again. The sample was
15 then grasped between the thumb and fore finger on opposite edges and twisted as far as possible to wring oil from the sample. The oil was allowed to drain while the sample was twisted. When no further oil drained from the twisted sample the sample was untwisted. The sample was weighed to the nearest 0.01 gram and
20 the mop up capacity was determined.

The capacity of the material to absorb and mop up water was determined by the same procedures utilized for oil except that distilled water was used instead of oil.

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The drape stiffness measurements were performed using a Shirley Stiffness Tester available from Shirley Developments Limited, Manchester, England. Test results were obtained essentially in accordance with ASTM Standard Test D 1388 except that the sample
30 size was 1 inch X 8 inches with the larger dimension in the direction being tested.

The levels of (1) extractables in isopropyl alcohol, 1,1,1-trichloroethane and distilled water and (2) the concentration of
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sodium ions was determined by the following procedure. Duplicate samples of the wipes weighing approximately 2 grams were refluxed for 4 hours in 200 mL of solvent using a soxhlet extraction apparatus. The solvent was evaporated to dryness and the percent extractables was calculated by determining the difference in the weight of the container before and after evaporation. The percent extractables is expressed as weight percent of the starting material. The quantity of sodium in the sample was determined by measuring the concentration of sodium ions in water obtained from the soxhlet extraction apparatus after the water extractables test. A Perkin-Elmer Model 380 atomic absorption spectrophotometer was used to measure the sodium ion concentration in the water.

The abrasion resistance of the material was determined essentially in accordance with British Standard Test Method 5690: 1979 with the following changes: (1) the abrasion machine used was available under the trade designation Martindale Wear and Abrasion Tester Model No. 103 from Ahiba-Mathis, Charlotte, North Carolina; (2) the samples were subjected to 100 abrasion cycles under a pressure of 1.3 pounds per square inch (psi) or 9 kilopascals (kPa); (3) a 1.5 inch diameter abradant was a cut from a 36 inch X 4 inch X 0.050 (± 0.005) inch piece of glass fiber reinforced silicone rubber having a surface hardness of 81A Durometer, 81 \pm 9 Shore A available from Flight Insulation Incorporated, Marietta, Georgia, distributors for Connecticut Hard Rubber; and (4) the samples were examined for the presence of surface fuzzing (fiber lofting), pilling, roping, or holes. The samples were compared to a visual scale and assigned a wear number from 1 to 5 with 1 indicating little or no visible abrasion and 5 indicating a hole worn through the sample.

EXAMPLE 1

A mixture of about 50 percent by weight hardwood pulp available from the Weyerhaeuser Company under the trade designation Grade

Regular and about 50 percent by weight uncrimped polyester staple fiber (1.5 denier x 12 mm), was dispersed to a consistency of about 0.5 percent by weight solids and then formed into handsheets of about 75 gsm on a standard 94 x 100 mesh plastic screen.

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A manifold available from Honeycomb Systems, Incorporated was utilized to entangle the handsheets. The handsheets were transferred to a standard 100 x 92 mesh stainless steel wire. The manifold was positioned approximately one-half (1/2) inch above the stainless steel wire mesh. The manifold contained a strip having 0.005 inch diameter orifices, 40 holes per inch and 1 row of holes. The strip was inserted into the manifold with the conical shaped holes diverging in the direction of the wire. Entanglement was performed with the handsheet travelling at a speed of about 20 meters per minute.

The handsheets were entangled at pressures of 200, 400, 600, 800, 1200 and 1400 psi on one side of the sheet and at pressures of 1200 and 1400 psi on the opposite side of the sheet. The flow rate of the entangling water was 1.054 cubic meters per hour per inch of strip. The entangled sheets were air dried at ambient temperature. The dried material had a basis weight of about 70 gsm.

Samples of the entangled material having a width of about 4 inches were tested using an Intellect II tensile testing apparatus available from Thwing Albert and an Instron Model 1122 Universal Testing Instrument, each having a 3 inch jaw span and a crosshead speed of about 12 inches per minute. Values for Peak Load, Peak EA, Peak Percentage Elongation, TEA and Total Percentage Elongation for the dry samples are reported in Table 1 for the machine direction and the cross-machine direction. Similar data was collected for wet samples in the machine direction only and is also reported in Table 1.

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EXAMPLE 2

5 A mixture of about 20 percent by weight hardwood pulp available from the Weyerhaeuser Company under the trade designation Grade Regular, about 40 percent by weight uncrimped polyester staple fiber (1.5 denier x 12 mm) and about 40 percent by weight uncrimped rayon staple fiber (1.5 denier x 12 mm) was dispersed and then formed into handsheets of about 75 gsm on a standard 94 x 100 mesh plastic screen.

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The handsheet was entangled using the equipment and procedure of Example 1 on a standard 100 x 92 mesh stainless steel wire at pressures of 600, 900, 1200 and 1500 psi on one side of the sheet and at pressures of 1200 and 1500 psi on the opposite side of the
15 sheet. The flow rate of the entangling water was 0.808 cubic meters per hour per inch of strip. The entangled sheets were air dried at ambient temperature. The dried material had a basis weight of about 73 gsm.

20 Samples of the entangled material having a width of about 4 inches were tested using the equipment and procedures of Example 1. Values for Peak Load, Peak EA, Peak Percentage Elongation, TEA and Total Percentage Elongation for the dry samples are reported in Table 2 for the machine direction and the cross-
25 machine direction.

EXAMPLE 3

A mixture of about 18.5 percent by weight hardwood pulp available from the Weyerhaeuser Company under the trade designation Grade
30 Regular, about 78.5 percent by weight uncrimped polyester staple fiber (1.5 denier x 12 mm) and about 3 percent by weight polyvinyl alcohol binder fiber was dispersed and then formed continuously onto a foraminous surface at about 60 gsm. The web was formed utilizing a continuous inclined wire paper making

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machine. The web was dried over a series of steam heated cans. Polyvinyl alcohol was added to facilitate reeling and handling.

The dried web was re-wetted and then entangled using the equipment and procedure of Example 1 on a standard 100 x 92 m sh
5 stainless steel wire employing 6 passes at pressures of 1800 psi on each side of the sheet. The flow rate of the entangling water was 2.04 cubic meters per hour per inch of strip. The entangled sheets were air dried at ambient temperature. The dried material had a basis weight of about 53 gsm.

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Samples of the entangled material having a width of about 4 inches were tested using the equipment and procedures of Example 1. Values for Peak Load, Peak EA, Peak Percentage Elongation, TEA and Total Percentage Elongation for the dry samples are
15 reported in Table 3 for the machine direction and the cross-machine direction.

EXAMPLE 4

A mixture of about 19 percent by weight hardwood pulp available
20 from the Weyerhaeuser Company under the trade designation Grade Regular, about 39 percent by weight uncrimped polyester staple fiber (1.5 denier x 12 mm), about 39 percent by weight uncrimped rayon staple fiber (1.5 denier x 12 mm) and about 3 percent by weight polyvinyl alcohol binder fiber was dispersed and then
25 formed continuously onto a foraminous surface at about 60 gsm. The web was formed utilizing a continuous incline wire paper making machine. The web was dried over a series of steam heated cans. Polyvinyl alcohol was added to facilitate reeling and handling.

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The dried web was pre-wetted and then entangled using the equipment and procedure of Example 1 on a standard 100 X 92 m sh stainless steel wire. Pre-wetting was done on one side at pressures of 200, 400 and 600 psi. Entangling on that side was
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performed at pressures of 800, 1000, 1200 and three passes at 1500 psi. The other side of the material was entangled by 3 passes at 1500 psi. The entangled sheets were air dried at ambient temperature. The dried material had a basis weight of about 53 gsm.

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Samples of the dried and the entangled material having a width of about 4 inches were tested using an Intellect II tensile testing apparatus with a 3 inch jaw span and a crosshead speed of about 10 inches per minute. Values for Peak Load, Peak EA and Peak Strain are reported in Table 4 for the machine direction and the cross-machine direction for dry samples. Similar results are also reported in Table 4 for wet samples.

For comparative purposes, Table 5 lists the Thickness Index, Isotropic Strength Index, abrasion test results, and drape stiffness test results for the entangled material of Examples 2, the entangled and unentangled material of Example 4, and two commercially available materials which can be used for wiping. Wiper A is a hydraulically entangled nonwoven material having the trade designation Sontara, grade 8005 available E.I. duPont de Nemours and Company. Wiper B is made from a wood pulp/staple fiber blend formed by laying a wood pulp web over a staple fiber web and then hydroentangling the webs. Wiper B has the trade designation Mohair Bleu and is available in France from Maury of Nantes, France and from Sodave of Angers, France. Table 5 also lists the thickness index and the isotropic strength index for the identified materials.

As can be seen from Table 5, the hydroentangled materials from Examples 2 and 4 have a greater thickness index than the unentangled material of Example 4, Wiper A and Wiper B. The materials from Examples 2 and 4 also have a greater isotropic strength index than Wipers A and B.

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Tabl 6 provid s results of testing f r the absorption rate, total abs rptiv capacity and mop-up capacity of th material from Example 4 for oil and water. The material f ExAMPL 2 had a total absorptive capacity and mop-up capacity for both oil and water which is significantly greater than the values for Wiper B.

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Tables 7, 8 and 9 provide test results for the materials of the present invention and for various other wipers that are commercially available in Europe. Wiper CW1 is made of a meltblown polypropylene fabric. Wiper CW2 is a laminate of spunbonded polypropylene/meltblown polypropylene/spunbond d polypropylene. The wiper available under the trade designation MIRACLE WIPES is made of hydroentangled staple and cellulosic fibers. The wiper available under the trade designation CLEAN ROOM WIPERS is made of wet formed staple and cellulosic fib rs. 15 The wiper available under the trade designation DURX is made f hydroentangled staple and cellulosic fibers. The wiper available under the trade designation LABX is made of wet-formed staple and cellulosic fibers. The wiper available under the trade designation TEXWIPE is made of a 100 percent cotton woven fabric. 20 The wiper available under the trade designation MICRONWIPE is made of hydroentangled staple and cellulosic fibers. The wiper available under the trade designation TEXBOND is made of a spunbonded nylon fabric. The wiper available under the trade designation TECHNI-CLOTH is made of hydroentangled staple and 25 cellulosic fibers.

For comparative purposes, Table 7 lists the results of extractable tests and sodium ion tests for the material of Example 2 and for some of the above-mentioned wipers. Also 30 included in Table 7 are results for two materials made according Example 2. Material H contains about 80 percent by weight rayon staple fibers and about 20 percent by weight wood pulp. Material F contains about 80 percent by weight polyester staple fibers and about 20 percent by weight wood pulp. Table 8 lists the results

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of electrical charge dissipation tests for the material of Example 2, Wiper A and for some of the above-mentioned wipers. Table 9 lists the results of ClimeTM lint tests for the materials from Example 2, the entangled and untangled material from Example 4, Wiper A, and for some of the above-mentioned
5 wipers.

As shown in Table 7, the materials of the present invention have levels of extractables which compare favorably with many commercial wipers. From Table 8, it can be seen that the
10 materials of the present invention without any anti-static treatment have a static decay which is comparable with many commercial wipers. From Table 9, it can be seen that the materials of the present invention have relatively low lint levels and compare favorably with many commercial wipers.

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Thus, it is apparent that the present invention provides a wiper that satisfies problems associated with previous wipers. While the invention has been described in conjunction with specific embodiments, the disclosed embodiments are intended to illustrate
20 and not to limit the invention. It is understood that those of skill in the art should be capable of making numerous modifications without departing from the true spirit and scope of the invention.

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TABLE 1

<u>DRY</u>	<u>MACHINE DIRECTION</u>	<u>CROSS- MACHINE DIRECTION</u>
Peak Load (g)	11,677	8699
Peak Energy Absorbed (kg-mm)	106	62
Peak Strain (%)	68.5	53.8
Total Energy Absorbed (kg-mm)	198	146
Total Strain (%)	131	122

WET

Peak Load (g)	7214
Peak Energy Absorbed (kg-mm)	117
Peak Strain (%)	120
Total Energy Absorbed (kg-mm)	196
Total Strain (%)	217

TABLE 2

<u>DRY</u>	<u>MACHINE DIRECTION</u>	<u>CROSS- MACHINE DIRECTION</u>
Peak Load (g)	9125	8749
Peak Energy Absorbed (kg-mm)	55	55
Peak Strain (%)	46	49
Total Energy Absorbed (kg-mm)	114	110
Total Strain (%)	100	104

TABLE 3

<u>DRY</u>	<u>MACHINE DIRECTION</u>	<u>CROSS- MACHINE DIRECTION</u>
Peak Load (g)	5035	4081
Peak Energy Absorbed (kg-mm)	63	71
Peak Strain (%)	89	118
Total Energy Absorbed (kg-mm)	124	99
Total Strain (%)	174	160

TABLE 4

<u>DRY</u>	<u>MACHINE DIRECTION</u>	<u>CROSS- MACHINE DIRECTION</u>
Peak Load (g)	4529	4133
Peak Energy Absorbed (kg-mm)	.83	79
Peak Strain (%)	39	49

<u>WET</u>	
Peak Load (g)	4014
Peak Energy Absorbed (kg-mm)	62
Peak Strain (%)	37

TABLE 5

	<u>EXAMPLE 2</u>	<u>EXAMPLE 4</u>	<u>BASE SHEET</u> <u>EXAMPLE 4</u>	<u>WIPER A</u>	<u>WIPER B</u>
Isotropic Strength Index	1.04	1.096	1.0	2.37	1.45
Thickness (mm)	0.79	0.73	0.36	0.44	0.31
Basis Weight (gsm)	73	53	60	65	75
Thickness Index	0.011	0.014	0.006	0.007	0.004
Drape Stiffness (cm)					
Side 1		3.6		3.4	7.7
Side 2		3.2		2.8	4.8
Martindale Abrasion Rating					
Side 1	1	2		2	3
Side 2	1	2		1	1

TABLE 6WATEREXAMPLE 4WIPE B

Absorption Rate (sec.)	1.0	<1
Total Absorptive Capacity (%)	553	347
Mop-Up Capacity (%)	258	151

OIL

Absorption Rate (sec.)	9.0	7.0
Total Absorptive Capacity (%)	596	230
Mop-Up Capacity (%)	250	33

TABLE 7EXTRACTABLES

<u>PRODUCT/CODE</u>	<u>% in isopropyl alcohol</u>	<u>% in 1,1,1-tri- chloroethane</u>	<u>% in hot water</u>	<u>hot water leachable sodium (ppm)</u>
CW1	0.8	0.7	2.5	43
CW2	0.8	3.5	0.2	47
Miracle Wipes® 1003	<0.1	1.7	0.2	20
Clean Room Wipers® 8025	2.1	1.6	1.0	391
Durx® 670	<0.1	<0.1	0.3	41
Durx 770	0.2	0.1	0.4	177
Labx 123	<0.1	<0.1	1.1	206
Texwipe™ 309	0.3	<0.1	3.1	47
Micronwipe® 500	<0.1	<0.1	3.4	1030
Texbond™ 909	5.4	3.4	0.3	1640
Techni-Cloth® 609	<0.1	4.3	1.4	43
Techni-Cloth® II 1009	<0.1	0.4	2.5	43
Example 2	0.1	0.1	1.5	126
Wiper Material H	0.2	0.1	1.0	133
Wiper Material F	0.2	0.1	0.6	116

TABLE 8ELECTRICAL PROPERTIES

<u>PRODUCT/CODE</u>	<u>static decay</u> <u>(sec)</u>
CW1	14.7
CW2	19.8
Miracle Wipes® 1003	0.3
Clean Room Wipers® 8025	4.5
Durx® 670	5.8
Labx® 123	1.3
Texwipe™ 309	1.1
Micronwipe® 500	0.9
Texbond™ 909	6.5
Techni-Cloth® 609	15.2
Techni-Cloth® II 1009	1.6
Example 2	7.0
Wiper A	No Dissipation
Wiper B	3.6

Notes: 1. Higher static decay times indicate increased tendency for static charge accumulation.

TABLE 9CLIMET LINE (# PARTICLES)

<u>PRODUCT/CODE</u>	<u>10 u</u>	<u>0.5 u</u>
CW1	0.4	112
CW2	0.1	9
Miracle Wipes® 1003	0.2	56
Clean Room Wipes® 8025	0.2	4
Drux® 670	0.4	442
Labx® 123	0.4	428
Texwipe™ 309	2.6	5130
Micronwipe® 500	0.7	498
Texbond™ 909	0.0	7
Techni-Cloth® 609	0.6	358
Techni-Cloth® II 1009	0.4	8
Example 2	2	65
Example 4 (Entangled)	0.8	76
Example 4 (Base Sheet)	2	72
Wiper A	0.8	51
Wiper B	0.2	286
Example 1	0.2	328

What is claimed is:

1. A hydraulically entangled coherent fibrous structure comprising:
 - from about 10 to about 50 percent by weight wood pulp fibers; and
 - 5 from about 50 to about 90 percent by weight of staple fibers; and
 - wherein said structure has a basis weight of from about 30 gsm to about 150 gsm and a thickness index of at least about 0.008.
2. A hydraulically entangled coherent fibrous structure comprising:
 - from about 10 to about 50 percent by weight wood pulp fibers; and
 - 5 from about 50 to about 90 percent by weight of staple fibers; and
 - wherein said structure has a basis weight of from about 30 gsm to about 150 gsm and an isotropic strength index less than about 1.5.
3. A hydraulically entangled coherent fibrous structure comprising:
 - from about 0 to about 50 percent by weight wood pulp fibers; and
 - 5 from about 50 to about 100 percent by weight of staple fibers; and
 - wherein said structure has a basis weight of from about 30 gsm to about 150 gsm and a thickness index of at least about 0.008.
4. A hydraulically entangled coherent fibrous structure comprising:
 - from about .0 to about 50 percent by weight wood pulp fibers; and

5 from about 50 to about 100 percent by weight of staple fibers; and

wherein said structure has a basis weight of from about 30 gsm to about 150 gsm and an isotropic strength index less than about 1.5.

5. The structure of claim 1 wherein the staple fibers have a denier in the range of about 0.7 to about 3 and an average length in the range of about 5 mm to about 18 mm.

6. The structure of claim 1 wherein said staple fibers comprise one or more of rayon, cotton, polyesters, polyolefins, and polyamides.

7. The structure of claim 1 wherein the material has an oil absorption capacity of at least about 300 percent.

8. The structure of claim 1 wherein the material has a water absorption capacity of at least about 375 percent.

9. The structure of claim 1 wherein the material has a sodium ion content of less than about 150 parts per million.

10. A hydraulically entangled coherent fibrous structure comprising:

from about 10 to about 50 percent by weight wood pulp fibers; and

5 from about 50 to about 90 percent by weight of staple fibers; and

wherein said structure has a basis weight of from about 30 gsm to about 150 gsm, a thickness index of at least about 0.008 and a water absorption capacity of at least about 375 percent.

11. A hydraulically entangled coherent fibrous structure comprising:

from about 10 to about 50 percent by weight wood pulp fibers; and

5 from about 50 to about 90 percent by weight of staple fibers; and

wherein said structure has a basis weight of from about 30
gsm to about 150 gsm, an isotropic strength index less than
about 1.5 and an oil absorption capacity of at least about 300
10 percent.

12. A hydraulically entangled coherent fibrous structure comprising:

from about 0 to about 50 percent by weight wood pulp fibers; and

5 from about 50 to about 100 percent by weight of staple fibers; and

wherein said structure has a basis weight of from about 30
gsm to about 150 gsm, a thickness index of at least about 0.008
and a water absorption capacity of at least about 375 percent.

13. A hydraulically entangled coherent fibrous structure comprising:

from about 0 to about 50 percent by weight wood pulp fibers; and

5 from about 50 to about 100 percent by weight of staple fibers; and

wherein said structure has a basis weight of from about 30
gsm to about 150 gsm, an isotropic strength index less than about
1.5 and an oil absorption capacity of at least about 300
10 percent.

14. The structure of claim 10 wherein the staple fibers have a
denier in the range of about 0.7 to about 3 and an average length
in the range of about 5 mm to about 18 mm.

15. The structure of claim 10 wherein said staple fibers comprise one or more of rayon, cotton, polyester, polyolefins, and polyamides.

16. The structure of claim 10 wherein the material has a sodium ion content of less than about 150 parts per million.



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(21) International Application Number: PCT/US89/04292 (22) International Filing Date: 29 September 1989 (29.09.89) (30) Priority data: 253,805 5 October 1988 (05.10.88) US (71) Applicant: KIMBERLY-CLARK CORPORATION [US/ US]; 401 North Lake Street, Neenah, WI 54956 (US). (72) Inventors: WATTS, Hugo, P. ; 6 Orwell Close, Larkfield, Kent ME20 6UP (GB). WATKINS, Sharon, L. ; 2300 Riverwood Lane, Apartment E, Roswell, GA 30075 (US). EVERHART, Cherie, H. ; 100 Shadow Springs Drive, Alpharetta, GA 30201 (US). VANDER WIELEN, Mi- chael, J. ; 360 Roswell Farms Road, Roswell, GA 30075 (US).		(74) Agent: SIDOR, Karl, V.; Kimberly-Clark Corporation, 401 North Lake Street, Neenah, WI 54956 (US). (81) Designated States: AT (European patent), AU, BE (Euro- pean patent), CH (European patent), DE (European pa- tent), DK, FI, FR (European patent), GB (European pa- tent), IT (European patent), JP, KR, LU (European pa- tent), NL (European patent), NO, SE (European patent). Published <i>With international search report.</i> <i>Before the expiration of the time limit for amending the</i> <i>claims and to be republished in the event of the receipt of</i> <i>amendments.</i> (88) Date of publication of the international search report: 07 February 1991 (07.02.91)
(54) Title: HYDRAULICALLY ENTANGLED WET LAID BASE SHEETS FOR WIPERS (57) Abstract A cloth-like nonwoven material useful for wiping and having strength, toughness, abrasion resistance and resistance to cer- tain solvents and/or chemicals is made from mixtures of wood pulp and staple fibers randomly distributed and hydraulically en- tangled with each other to form a coherent entangled fibrous structure having a thickness index of at least about 0.008 and a ratio of machine direction strength to cross machine direction strength of at least about 1.5.		

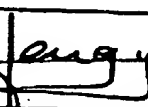
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A	--- DATABASE WPIL, no.86-192792 Derwent Publications Ltd, London, GB, & JP-A-61124610(Teijin KK) 12 June 1986 see the whole abstract	1-16
A	--- EP,A,0108621 (E.I.DU PONT DE NEMOURS AND CO.) 16 May 1984 see the whole document	
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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A-0308320	22-03-89	US-A- 4808467	28-02-89
		JP-A- 1111056	27-04-89
EP-A-0108621	16-05-84	US-A- 4442161	10-04-84
		AU-B- 556706	13-11-86
		AU-A- 2085283	10-05-84
		CA-A- 1247346	27-12-88
		JP-A- 59094659	31-05-84
US-A-3493462	03-02-70	LU-A- 47555	10-06-66
		CH-B- 465545	
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